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ADHESION IN ROCKS

BUREAU OF MINES

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FINAL TECHNICAL REPORT

Bureau of Mines In-House Research
Adhesion in Rocks

Sponsored by

Advanced Research Projects Agency
ARPA Order No. 1579, Amendment No. 3
Program Code No. 2F10

TWIN CITIES MINING RESEARCH CENTER

Thomas C. Atchison, Research Director

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13. ABSTRACT

The objective was to ascertain the magnitude of forces responsible for coherency of rock by quantifying the strength of the attractive forces operating between minerals in rock. These forces oppose stresses set up in various rock fragmentation processes, hence strength measurements of these forces might prove useful in designing more efficient rock fragmentation methods.

Methods were developed for estimating strength of intergranular adhesion in rock. They involve selective extraction of 4.5 mm diameter, two-phase sample disks from rock and determination of the strength of the solid-solid interface. One method utilizes direct pull tests of the tensile strength of the interfaces, the other utilizes Brazilian tests of the extracted solid-solid interfaces. These techniques have been successfully used in studying quartz-feldspar interfaces from graphic and Rockville granites and pebble-matrix interfaces from Calumet conglomerate. Examination of bicrystals broken at the crystalline interfaces reveals that bonds responsible for this adhesion operate only over a portion of the interfacial area.

This work demonstrated that strength tests can be conducted on small selected areas, i.e., grain boundaries in rock. This permits a determination of small scale zones of strength or weakness which may be related to the overall strength of the rock.

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FINAL TECHNICAL REPORT

ADHESION IN ROCKS

by

George A. Savanick[1], Principal Investigator
Donald I. Johnson[2]

TECHNICAL REPORT SUMMARY

The purpose of this research is to ascertain the magnitude of the forces responsible for the coherency of rock by measuring the strength of the attractive forces operating to bind minerals together. These forces act in opposition to the stresses set up in various rock fragmentation processes, hence measurements of the strength of these attractive forces might prove useful in the design of more efficient methods of rock fragmentation.

The main outcome of this research has been the development of methods for estimating the strength of intergranular adhesion in rocks. These methods, which are outlined in detail in the "Experimental Procedure" section of the report, involve the selective extraction of a small (4.5 mm diameter, 1.7 mm thick) two-phase sample from the rock and a determination of the strength of the solid-solid interface. One of these methods utilizes direct pull tests of the tensile strength of the interfaces while the other utilizes indirect tensile (Brazilian) tests of the extracted solid-solid interfaces.

These techniques have been successfully applied to the study of quartz-feldspar interfaces separated from graphic granites and from the Rockville

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granite and to the study of pebble-matrix interfaces extracted from the Calumet conglomerate. The results of these tests are tabulated in the "Experimental Data" section of the report. These data indicate that:

1) Intermineralic interfaces can resist tensile stresses in excess of 1,000 psi, thus the minerals must be bonded to each other.
2) The adhesive strength of the grain boundaries is generally lower than the cohesive strength of the adjacent minerals.
3) The bond between the pebbles and the matrix in the Calumet conglomerate is stronger than the bond between quartz and feldspar in either the Rockville granite of the graphic granite.
4) Wide variability occurs in the magnitude of the individual strength determinations. This is probably a reflection of the nonuniform distribution of flaws in the rock.
5) The uniaxial pull test gives interfacial adhesive strength values approximately 20 percent lower than those given by the Brazilian test. Thus there appears to be a bias in the Brazilian test which favors higher values of tensile strength.

Examination of bicrystals broken at their grain boundary reveals that the bonds responsible for this adhesion operate over only a portion of the interfacial area.

The significance of this work is that it demonstrates that strength tests can be conducted on small selected areas, e.g. grain boundaries in rock. This permits a determination of small scale zones of strength or weakness which may be related to the overall strength of the rock.

Suggestions for future research on adhesion at grain boundaries in rocks are given in the final section of the report. These suggestions include the extension of adhesive strength testing to finer grained rocks, the development of methods of testing the shear strength of grain boundaries, and the application of selected area strength tests to drilling research.

INTRODUCTION

Rock fragmentation occurs when the applied energy overcomes the attractive forces holding the rock together. Characterization of these forces would seem to be a prerequisite for the optimization of the fragmentation process.

The chemical bonds responsible for the mutual attraction of atoms which gives rise to the cohesion of crystals have long been an object of study (1)[3]. Chemical bond strengths have been measured and are tabulated (1, 2). On the other hand, very little effort has been expended in understanding adhesion at phase boundaries in rock, i.e., the mechanism by which the constituent minerals are joined together at crystalline interfaces to form a coherent polycrystalline aggregate. Thus, prior to this research, no measurements had been made of the adhesive strength at crystalline interfaces in rock.

The objective of this research is to fill this void by developing a method of selected area tensile strength testing by which the tensile strength of crystalline interfaces can be measured and to present a tabulation and an interpretation of these data.

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[3] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

A publication resulting from this research project (3) contained the first reported measurements of the strength of crystalline interfaces in rock. The paucity of previous work is a reflection of the difficulties inherent in such measurements. The crystalline interface must be separated from the rock prior to any tensile strength testing. This can be very tedious, and in itself, might discourage investigators.

It must also be realized that pure adhesive failure is an idealization which can be approximated but cannot be attained. The requirement that the rupture must occur precisely at the atomic boundary between two phases renders pure adhesive failure very improbable. This is a problem that confronts any destructive testing of adhesive joints in the adhesives industry (4). Bits of one mineral will adhere to the other member of the broken bicrystal. This prohibits a direct measurement of the number of bonds which bind one mineral to the other, but it permits a determination of the areal extent of the bonding between the grains.

The limitations inherent in this type of experimentation tend to narrow the scope of the research. The tests were limited to planar interfaces in an attempt to eliminate the strengthening contribution from microscopic interlocking of phases. In addition, the difficulties of extraction and the errors in the tensile strength measurement increase as the size of the bicrystal decreases. In view of these difficulties, it was decided to limit consideration to relatively large (4.5 mm diameter) selected regions containing quartz-feldspar bicrystals extracted from pegmatites, and graphic granites, pebble-matrix interfaces extracted from a conglomerate.

This report contains a description of a method for extracting bicrystals from selected areas in rocks and two methods for determining the tensile strength at the crystalline interfaces. Adhesive strength data for crystalline interfaces extracted from graphic granites and the Rockville granite are tabulated and compared with the tensile strength of quartz and feldspar taken from the same rock. In addition, strength data for pebble-matrix interfaces extracted from a conglomerate are tabulated and compared with the strength of the adjacent pebbles and matrix. These measurements permit an assessment of the strength of adhesion at crystalline interfaces and a comparison with the cohesive strength of the constituent phases.

EXPERIMENTAL PROCEDURE

In adhesion technology, adhesion is defined (4) as the force per unit area required to separate two solids in contact. The magnitude of this stress can only be estimated from the results of destructive testing (5). The most easily interpreted measure of adhesion is the normal tensile force required for separation, hence it was decided to develop a method of selected area tensile strength testing to measure the adhesion at crystalline interfaces in rocks.

A successful method for selected area strength testing must provide for the extraction of planar intercrystalline boundaries from the rock and permit the separation of the joined crystals at the crystalline interface. A technique for selectively extracting boundaries has been developed and is outlined in stepwise fashion below.

(1) Rock samples are cut into 1/4 inch thick slabs.

(2) These slabs (fig. 1) are fastened to the surface of a block of soft wood with a fast drying epoxy cement. The wood surface is first sprayed with an aluminum-flake enamel in order to facilitate removal of the rock-epoxy ensemble to permit reuse of the wooden block.

(3) An area containing the trace of a planar crystalline interface is selected and removed by drilling with a Felker diamond core bit mounted in a Wilton drill press (fig. 2). It was found that 1/4 inch O.D. (6.4 mm) core drills worked well for extracting quartz-feldspar bicrystals from graphic granite and from the Rockville granite and pebble-matrix interfaces from the Calumet conglomerate. However, it is possible to selectively extract cores as small as 2.8 mm in diameter.

(4) Those portions of the crystalline interface which are nonplaner or off center are removed by grinding perpendicular to the cylinder axis with a thin section grinder (fig. 3).

Samples of these extracted interfaces were subjected to indirect tensile strength testing and direct pull strength testing. The procedure followed in the indirect tensile (Brazilian) testing of these interfaces is as follows:

FIGURE 1. - Slabs of Graphic Granite Cemented to a Wooden Block.

FIGURE 2. - Rock sample in place under a
diamond tipped core drill

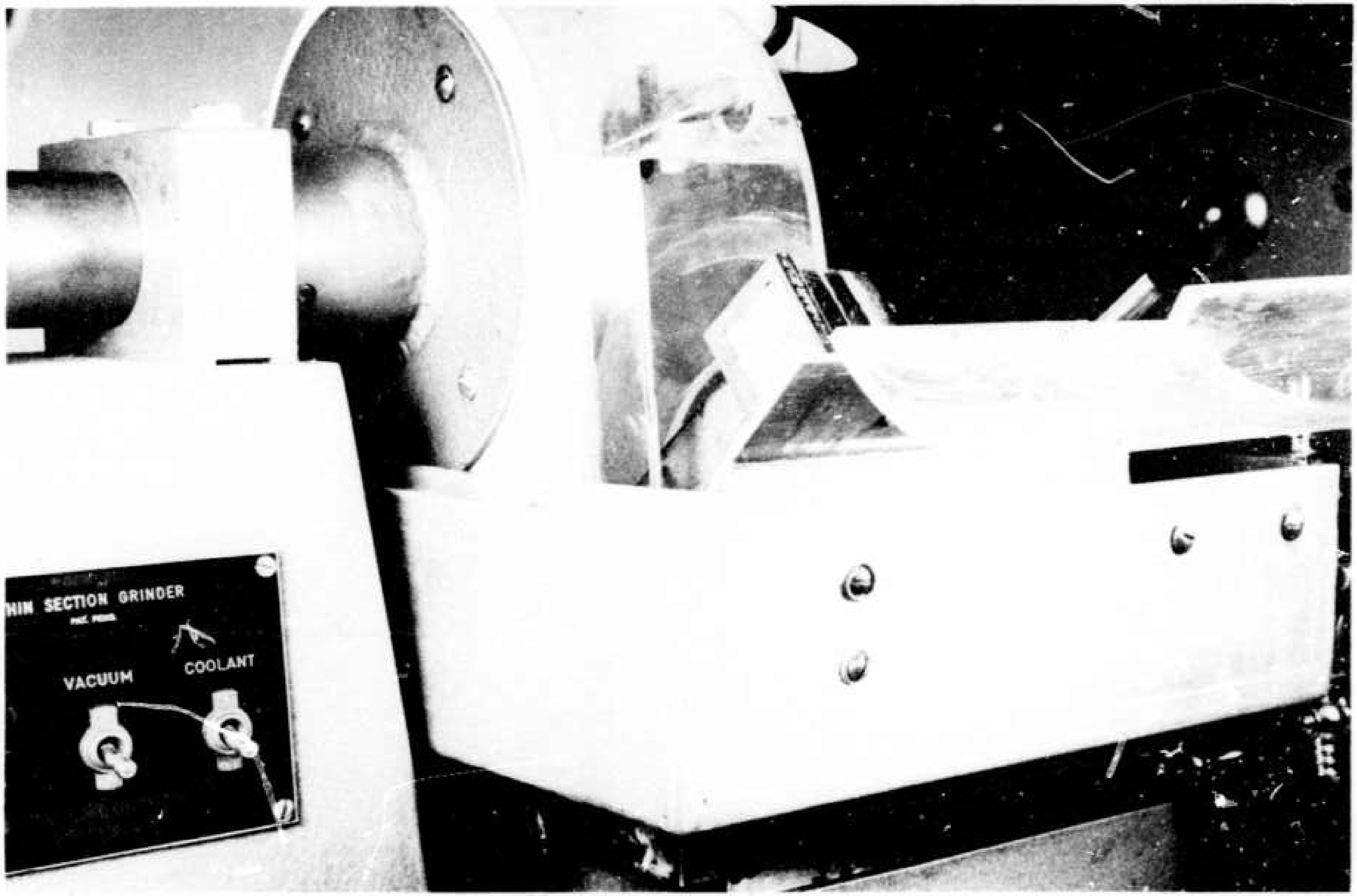

FIGURE 3. - Bicrystal Mounted on the Arm of a Thin Section Grinder.

(1) The diameter of the cylinder and the length of the cylinder axis is measured with calipers or a micrometer.

(2) The sample is placed in an Instron testing machine (fig. 4) and loaded in diametrical compression. The sample is oriented so that the stress is concentrated and the sample breaks at the crystalline interface.

(3) The tensile strength (S_t) of the intercrystalline boundary is calculated using the formula:

$$S_t = \frac{2L}{\pi d l}$$

where L is the load applied to the sample, d is the diameter of the sample, and l is the length of the cylinder axis.

The advantage of this technique is that it is designed to test the strength of solid disks, the sample configuration resulting from the extraction procedure and because it provides a convenient method for selectively concentrating stress along any disk diameter. Sample preparation is very simple and Brazilian tests can be performed more rapidly than pull tests because no adhesive is used to hold the sample to a loading jig and thus no curing time is required.

Although some researchers (7) are of the opinion that the diametral compression of a solid disk gives a good measure of the tensile strength of rocks, the Brazilian test has become suspect to other members of the rock mechanics community (8) as a method for determining tensile strength. This provided the impetus for the development (9) of uniaxial method for testing the tensile strength of interfaces between minerals in rocks by pulling normal to these interfaces.

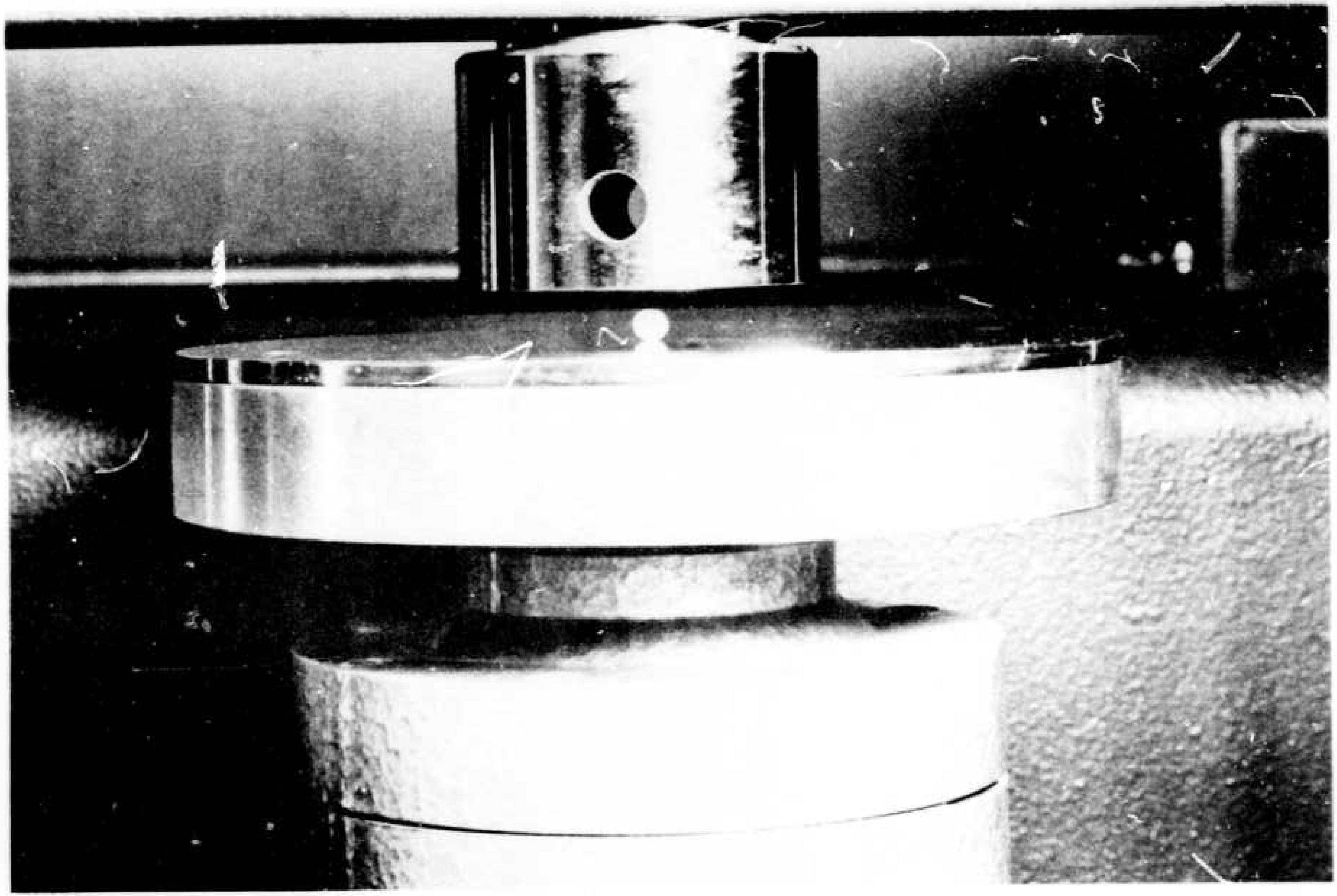

FIGURE 4. - Bicrystal in Place on the Load Cell of the Testing Machine.

The procedure followed in the uniaxial tensile strength testing of interfaces extracted from rocks is as follows:

(1) The sample is fastened to one half of a loading jig with a fast drying (five minute) epoxy cement. The loading jig is composed of two identical pieces made by cutting a brass rectangular parallelepiped in a direction parallel to its base thereby bisecting a cylindrical hole with a diameter the same as that of the sample (fig. 5).

(2) The sample-epoxy loading jig ensemble is clamped into the upper jaw set of an Instron TM-M testing instrument. Care must be taken to align the centerline of the loading jig with the centerline of the jaw set so that bending moments will not occur.

(3) The other half of the loading jig with unhardened epoxy cement in the semicircular hole is clamped into the bottom jaw set of the testing machine and the cement is placed in contact with the sample in the portion of the jig clamped into the upper jaw set. The cement is permitted to harden and thereby bond the small cylindrical sample to both sides of the loading jig (fig. 6).

(4) The sample is pulled apart and the load at failure is noted.

This technique yields data which are much easier to interpret and is capable of testing smaller samples than the Brazilian test, however it requires more time per test.

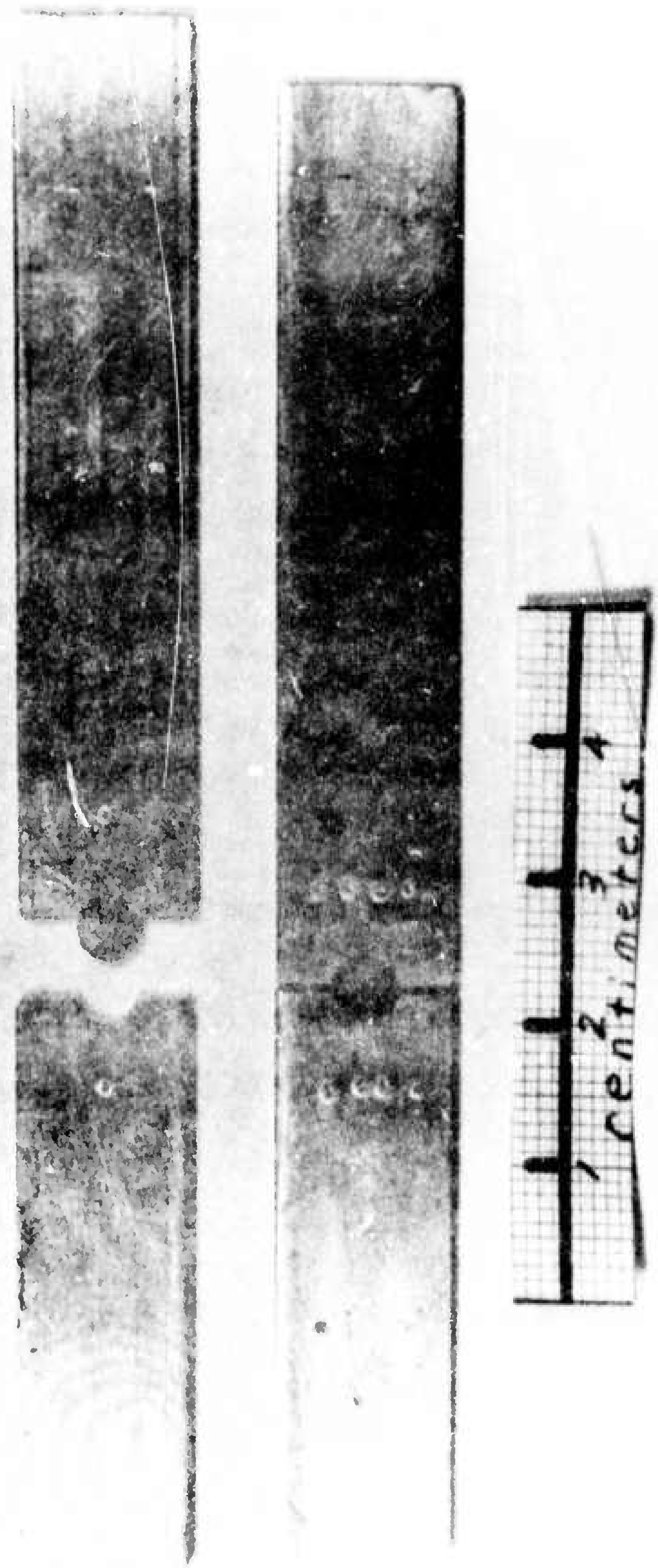

FIGURE 5 – Sample jig for direct pull tensile tests.

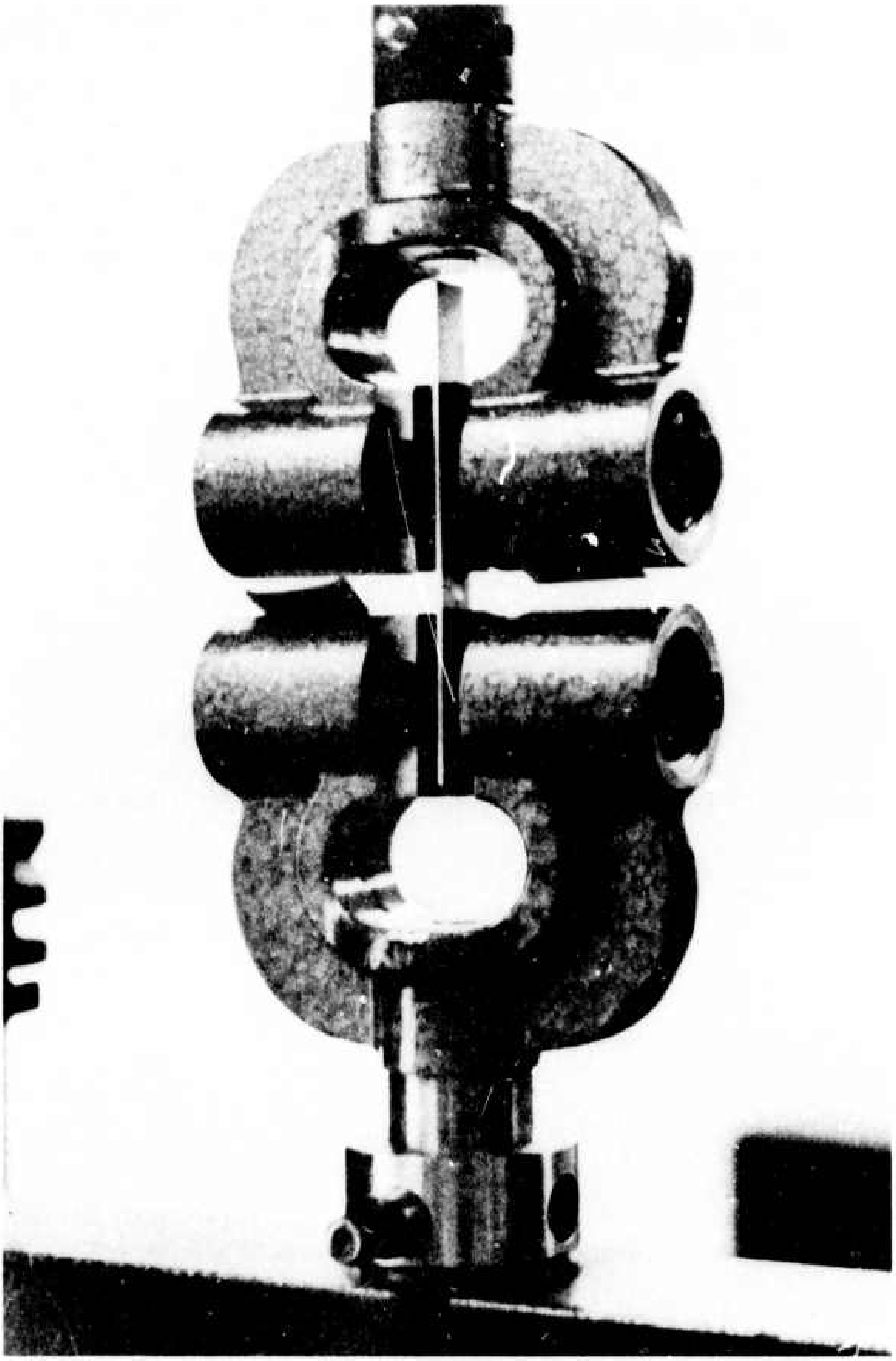

FIGURE 6. - Sample, epoxy, loading jig ensemble in place, in testing equipment.

The tensile tests result in bicrystal samples (fig. 7) that have separated at the crystalline interface and yield quantitative measures of the intergranular adhesive strength. Chemical bonding between the quartz and feldspar occurred over a small proportion of the total interfacial area.

EXPERIMENTAL DATA

The data generated by the techniques described above are tabulated in this section. These tables give the magnitude of the adhesive strength at interfaces between phases in rocks and permit the comparison of this strength with that of the phases adjacent to the interface.

Tables 1, 2, and 3 contain the results of indirect tensile (Brazilian) tests of selected areas in a graphic granite, the Rockville granite, and the Calumet conglomerate.

The results of the "t" (11) tests of significance for differences of means of various subsets of these data are contained in table 4.

These tables illustrate that:

1. The intermineralic interfaces have a significant tensile strength. This indicates that the minerals must have adhered to each other.
2. The adhesive strength at the intermineralic interface is significantly lower than the cohesive strength of the adjacent minerals.
3. Wide variability occurs in the magnitude of the individual determinations of strength.

The most serious problem encountered during the past year was the realization that the Brazilian test had become suspect as a true measure of the tensile strength. Many researchers (12) believe that the techniques can only be used as a relative measure of tensile strength and then only if

FIGURE 7. - Quartz-Feldspar Crystalline Interface With a Small Proportion of Bonded Area.

TABLE 1. - Tensile strength (S_t) of graphic granite (4.5 mm cores) as determined by the Brazilian test

Bicrystal data			Single crystal data			Single crystal data		
Quartz-feldspar interface	Brazilian test		Feldspar crystal	Brazilian test		Quartz crystal	Brazilian test	
	S_t, psi	S_t, MN/m^2		S_t, psi	S_t, MN/m^2		S_t, psi	S_t, MN/m^2
K721B	994	6.8	K721F	1,799	12.4	K724Q	3,384	23.3
K722B	2,035	14.0	K723F	2,319	16.0	K727Q	1,893	13.0
K723B	473	3.3	K724F	1,704	11.8	K7213Q	2,000	13.8
K724B	1,775	12.2	K725F	1,396	9.6	K7214Q	4,354	30.0
K725B	899	6.2	K726F	3,136	21.6	K7215Q	1,597	11.0
K726B	1,574	10.8	K728F	1,325	9.1	K7216Q	4,141	28.6
K727B	887	6.1	K729F	2,650	18.3	K7217Q	1,183	8.2
K728B	1,680	11.6	K7210F	4,023	27.7	K7218Q	887	6.1
K729B	2,343	16.2	K7211F	2,733	18.8	K7219Q	1,503	10.4
K7210B	2,390	16.5	K7212F	2,816	19.4	K7220Q	1,018	7.0
K7211B	769	5.3	K7213F	3,431	23.7	K7221Q	2,485	17.1
K7212B	580	4.0	K7214F	2,248	15.5	K7222Q	2,828	19.5
K7213B	828	5.7	K7215F	651	4.5	K7224Q	2,508	17.3
K7214B	1,195	8.2	K7216F	2,390	16.5	K7225Q	899	6.2
K7215B	532	3.7	K7217F	2,970	20.5	K7226Q	3,017	20.8
K7216B	1,006	6.9	K7218F	2,071	14.3	K7227Q	1,976	13.6
K7217B	1,136	7.8	K7219F	2,118	14.6	K7229Q	2,449	16.9
K7218B	734	5.0	K7220F	1,302	9.0	K7230Q	2,367	16.3
K7219B	2,189	15.1	K7221F	1,207	8.3	K7231Q	1,266	8.7
K7221B	1,325	9.1	K7222F	2,260	15.6	K7232Q	3,597	24.8
K7222B	734	5.0	K7223F	2,615	18.0			
			K7224F	5,822	40.1			
			K7225F	1,728	11.9			
			K7226F	2,639	18.2			
			K7227F	2,911	20.1			
			K7229F	3,502	24.2			
			K7230F	2,508	17.3			
Average	1,241	8.6	Average	2,454	16.9	Average	2,267	15.6

TABLE 2. - Tensile strength (S_t) of Rockville granite (4.5 mm cores) as determined by the Brazilian test

Bicrystal data			Single crystal data			Single crystal data		
Quartz-feldspar interface	Brazilian test		Feldspar crystal	Brazilian test		Quartz crystal	Brazilian test	
	S_t, psi	S_t, MN/m^2		S_t, psi	S_t, MN/m^2		S_t, psi	S_t, MN/m^2
0721I	1,999	13.8	0721F	1,929	13.3	0722Q	2,852	19.7
0722I	1,810	12.5	0722F	1,810	12.5	0724Q	2,461	17.0
0723I	922	6.4	0723F	1,219	8.4	07[illegible]Q	2,899	20.0
0724I	946	6.5	0724F	2,000	13.8	07[illegible]6Q	2,781	19.2
0725I	1,124	7.8	0725F	3,242	22.4	0727Q	3,502	24.2
0728I	2,070	14.3	0727F	1,799	12.4	0728Q	2,047	14.1
P721I	1,455	10.0	0728F	2,272	15.7	0729Q	1,988	13.7
P723I	1,479	10.2	0729F	1,609	11.1	07211Q	1,751	12.1
P724I	1,467	10.1	07210F	3,088	21.3	07212Q	2,236	15.4
P727I	674	4.6	07213F	3,408	23.5	07213Q	1,704	11.8
P728I	1,384	9.5	07215F	3,775	26.0	07214Q	1,775	12.2
P729I	2,367	16.3	07216F	1,739	12.0	07215Q	1,574	10.8
P7211I	1,467	10.1	07217F	2,118	14.6	07217Q	5,396	37.2
P7212I	1,586	10.9	07218F	2,071	14.3	07218Q	1,834	12.6
P7213I	1,917	13.2	07219F	1,822	12.6	07223Q	1,941	13.4
P7216I	2,520	17.[illegible]	07220F	2,485	17.1	07224Q	1,373	9.5
P7217I	355	2.4	07221F	2,426	16.7	07226Q	627	4.3
P7218I	935	6.4	07222F	2,012	13.9	07227Q	2,059	14.2
P7219I	2,059	14.2	07225F	4,177	28.8	07228Q	3,053	21.0
P7222I	1,266	8.7	07226F	1,278	8.8			
			07229F	1,810	12.5			
			07230F	2,189	15.1			
			07231F	2,154	14.8			
			07232F	3,112	21.5			
			07233F	2,307	15.9			
			07237F	2,142	14.8			
			07238F	3,195	22.0			
			07239F	1,065	7.3			
			07240F	1,633	11.3			
Average	1,490	10.3	Average	2,272	15.7	Average	2,308	15.9

TABLE 3. - Tensile strength (S_t) of Calumet conglomerate (4.5 mm cores) as determined by the Brazilian test

Pebble-Matrix interface	Brazilian test S_t psi	Brazilian test S_t MN/m²	Pebble	Brazilian test S_t psi	Brazilian test S_t MN/m²	Matrix	Brazilian test S_t psi	Brazilian test S_t MN/m²
L721I	2,355	16.2	L721P	3,763	26.0	L721M	1,941	13.4
L722I	2,899	20.0	L722P	4,059	28.0	L722M	2,662	18.4
L723I	2,307	15.9	L723P	4,922	33.9	L723M	899	6.2
L724I	733	5.0	L724P	2,686	18.5	L724M	2,757	19.0
L725I	2,461	17.0	L725P	4,047	27.9	L725M	4,603	31.7
L727I	2,331	16.1	L726P	5,112	35.2	L726M	3,431	23.7
L728I	2,710	18.7	L727P	2,130	14.7	L727M	2,729	19.2
L7210I	1,609	11.1	L728P	3,467	23.9	L729M	2,568	17.7
L7211I	4,201	29.0	L729P	4,390	30.3	L7210M	3,739	25.8
L7212I	2,710	18.7	L7210P	4,283	29.5	L7211M	2,023	13.9
M721I	1,408	9.7	L7211P	4,970	34.3	L7212M	4,141	28.6
M723I	2,579	17.8	L7212P	7,111	49.0	L7213M	3,621	25.0
M724I	2,485	17.1	L7213P	2,899	20.0	L7214M	2,579	17.8
M725I	1,313	9.0	L7214P	3,715	25.6	L7215M	2,603	17.9
M727I	2,958	20.4	L7215P	3,905	26.9	L7216M	2,508	17.3
N722I	2,012	13.9	L7216P	6,437	44.4	L7217M	2,284	15.7
N723I	2,307	15.9	L7217P	5,064	34.9	L7218M	3,337	23.0
N726I	2,544	17.5	L7218P	4,070	28.1	L7219M	1,881	13.0
N727I	3,100	21.4	L7219P	5,715	39.4	L7220M	2,000	13.8
N7213I	2,840	19.6	L7220P	3,372	23.2	L7221M	2,532	17.5
			L7221P	4,082	28.2	L7223M	4,054	28.0
			L7222P	3,846	26.5	L7224M	4,295	29.6
			L7223P	6,827	47.1	L7225M	1,716	11.8
			L7224P	4,603	31.7	L7227M	3,479	24.0
			L7225P	3,810	26.3	L7228M	2,840	19.6
			L7226P	2,615	18.0	L7229M	1,432	9.9
			L7227P	6,271	43.2	L7230M	1,361	9.4
			L7228P	6,271	43.2	L7231M	2,946	20.3
			L7229P	2,911	20.1	L7232M	2,367	16.3
			L7230P	4,378	30.2	L7233M	3,041	21.0
			L7231P	3,112	21.5	L7234M	3,775	26.0
			L7232P	4,615	31.8	L7235M	3,763	26.0
						L7236M	5,585	38.5
						L7237M	2,532	17.5
Average	2,393	16.5	Average	4,350	30.0	Average	2,885	19.9

TABLE 4. - Results of "t" tests of significance for differences of means of various subsets of data in tables 1, 2, 3

Comparison of Brazilian tests for	t statistic	Degrees of freedom	Probability that population means are different 1.0 = certainty	Means significantly different at 95% confidence level
Q-F Interfaces with F Graphic Granite	4.78718	46	> .995	Yes
Q-F Interfaces with Q Graphic Granite	3.86676	39	> .995	Yes
Q with F	0.61200	45	0.7	No
Q-F Interfaces with F Rockville Granite	3.89330	47	> .995	Yes
Q-F Interfaces with Q Rockville Granite	3.14889	37	> .995	Yes
Q with F Rockville Granite	-0.14128	46	0.50	No
P-M Interfaces with P Calumet Conglomerate	6.30570	50	> .995	Yes
P-M Interfaces with M Calumet Conglomerate	1.89152	52	< 0.55	No
P with M Calumet Conglomerate	-5.25777	64	> .995	Yes

samples of identical dimensions are used for comparison. Some variation in sample thickness has occurred in the earlier reported (3) data, so these tests were redone using samples having precisely the same dimensions. These results are given in tables 1, 2, and 3 and are summarized in table 5 where they can be compared with the earlier data. Note that the new data (those taken from samples with standardized dimensions of 4.5 mm diameter and 1.7 mm thickness) compare favorably with those reported previously (5 mm disk diameter).

A uniaxial pull test was developed to give an unequivocal method for testing the absolute tensile strength of crystalline interfaces. This technique was applied to selected areas in graphic granite, the Rockville granite, and the Calumet conglomerate. The results of these tests are tabulated in tables 6, 7, and 8 and are compared with the Brazilian tests on the same rocks in table 9.

These test results corroborate the results of the Brazilian tests, i.e. they show that:

1. Intermineralic interfaces can resist tensile stresses over 1,000 psi, thus the minerals must be bonded to each other.

2. The adhesive strength at the grain boundaries is generally lower than the cohesive strength of the adjacent minerals.

3. The bond between the pebbles and the matrix in the Calumet conglomerate is stronger than the bonds between quartz and feldspar in either the Rockville granite or the graphic granite.

4. Wide variability occurs in the magnitude of the individual strength determinations. This is probably a reflection of the non-uniform distribution of flaws in the rock.

TABLE 5. - Tensile strength of selected areas in graphic granite, Rockville granite, and the Calumet conglomerate: comparison of earlier results with those from samples with standardized dimensions

Rock	Sample	Disk Diameter	Disk thickness	Number of samples tested	Average splitting strength (psi)
Graphic granite	Quartz	5.0 mm	1.8-3.3	2	1,810
do.	Feldspar	5.0 mm	1.8-3.3	20	1,930
do.	Qtz-feldspar bicrystal	5.0 mm	2.3-4.5	17	1,210
Graphic granite	Quartz	4.5 mm	1.7	20	2,267
do.	Feldspar	4.5 mm	1.7	30	2,454
do.	Qtz-feldspar bicrystal	4.5 mm	1.7	21	1,241
Rockville granite	Quartz	5.0 mm	0.9-2.3	13	1,960
do.	Feldspar	5.0 mm	1.5-2.6	17	2,160
do.	Qtz-feldspar	5.0 mm	1.6-2.3	16	1,510
Rockville granite	Quartz	4.5 mm	1.7	19	2,308
do.	Feldspar	4.5 mm	1.7	29	2,272
do.	Qtz-feldspar bicrystals	4.5 mm	1.7	6	1,478
Calumet conglomerate	Pebble-matrix interface	4.5 mm	1.7	15	2,820
do.	Pebble-matrix interface	5.0 mm	1.7-3.2	8	2,577
Calumet conglomerate	Pebble-matrix interface	4.5 mm	1.7	20	2,393
do.	Pebbles			32	4,358
do.	Matrix			34	2,885

TABLE 6. - Tensile strength of selected areas in graphic granite as determined by the direct pull method

Interfaces			Quartz			Feldspar		
Sample	MN/m^2	psi	Sample	MN/m^2	psi	Sample	MN/m^2	psi
K721SI	12.81	1,857	R722SQ	19.09	2,767	R721SF	7.69	1,114
K722SI	29.59	4,290	R723SQ	25.24	3,659	R722SF	14.73	2,136
K723SI	8.45	1,225	R724SQ	13.45	1,950	R723SF	13.84	2,006
K724SI	6.28	910	R727SQ	17.29	2,507	R724SF	7.04	1,021
R721SI	8.71	1,263	R728SQ	15.76	2,284	R725SF	14.73	2,136
R723SI	12.68	1,838	R729SQ	17.55	2,544	R726SF	9.74	1,411
R724SI	12.81	1,857	R7210SQ	21.91	3,176	R727SF	9.86	1,430
R726SI	5.89	854	S732SQ	5.38	780	R728SF	9.22	1,337
R72 SI	3.72	538	S733SQ	11.27	1,634	R729SF	9.86	1,430
S731SI	1.79	260	S734SQ	4.61	668	R7210SF	14.73	2,136
S732SI	4.87	705	S735SQ	3.20	464	R7211SF	8.97	1,300
S733SI	4.74	687	S736SQ	11.53	1,671	S732SF	3.20	464
S734SI	1.67	241				S733SF	6.15	891
S735SI	3.33	482				S734SF	6.79	984
S736SI	3.20	464				S735SF	3.59	520
S737SI	2.18	315				S736SF	7.30	1,058
S738SI	2.18	315				S737SF	2.82	408
S839SI	5.12	743				S738SF	2.69	390
S8310SI	2.69	390				S739SF	5.89	854
						S7310SF	6.28	910
						S7311SF	11.91	1,727
						S7312SF	4.36	631
						S7313SF	3.84	557
						S7314SF	5.12	743
						S7315SF	7.17	1,040
						S7316SF	5.00	724
						S7317SF	5.76	835
						S7318SF	4.48	650
						S7319SF	2.05	297
Average	6.98	1,012	Average	13.86	2,009	Average	7.41	1,074

TABLE 7. - Tensile strength of selected areas in the Rockville granite as determined by the direct pull method

Interfaces			Quartz			Feldspar		
Sample	MN/m^2	psi	Sample	MN/m^2	psi	Sample	MN/m^2	psi
O722SI	3.84	557	P721SQ	5.64	817	P721SF	5.64	817
P721SI	5.76	835	P725SQ	15.95	2,312	P724SF	13.45	1,950
P728SI	7.04	1,021	P726SQ	16.52	2,396	P726SF	16.01	2,321
P729SI	12.17	1,764	P727SQ	5.64	817	P727SF	17.81	2,581
P7210SI	14.35	2,080	P728SQ	18.32	2,656	P729SF	12.43	1,801
P7211SI	16.01	2,321	P729SQ	12.30	1,783	P7210SF	10.25	1.486
P7212SI	8.45	1,225	P7211SQ	20.37	2,953	P7213SF	10.52	1,523
P7216SI	15.88	2,303	P7212SQ	11.53	1,671	P7214SF	11.17	1,597
P7220SI	7.81	1,133	P7214SQ	1.41	204	P7215SF	14.35	2,080
P7222SI	5.12	743	P7215SQ	7.69	1,114	P7216SF	4.74	687
P7223SI	4.74	687	P7216SQ	9.61	1,393	P7218SF	7.04	1,021
P7224SI	7.05	1,021	P7217SQ	3.33	482	P7219SF	13.45	1,950
P7227SI	8.07	1,170	P7218SQ	19.85	2,879	P7220SF	8.84	1,281
P7229SI	4.61	668	P7219SQ	10.38	1,504	P7221SF	10.12	1,467
P7230SI	5.25	761	P7220SQ	9.61	1,393	P7222SF	12.30	1,783
P7231SI	6.41	928	P7221SQ	6.41	928	P7223SF	5.38	780
P7233SI	4.87	705	P7222SQ	4.87	705	P7224SF	7.94	780
P7234SI	6.15	891	P7223SQ	7.30	1,058	P7225SF	7.05	1,151
						P7226SF	11.91	1,727
						P7227SF	10.76	1,560
Average	7.98	1,156	Average	10.37	1,504	Average	10.56	1,530

TABLE 8. - Tensile strength of selected areas in the Calumet conglomerate as determined by the direct pull method

Interfaces			Pebbles			Matrix		
Sample	MN/m^2	psi	Sample	MN/m^2	psi	Sample	MN/m^2	psi
M721SI	1.66	241				Q721SM	12.04	1,746
M722SI	23.96	3,474				Q723SM	16.27	2,359
M723SI	14.60	2,118				Q724SM	17.93	2,600
M724SI	18.58	2,693				Q725SM	19.73	2,860
M726SI	20.24	2,935				Q726SM	11.40	1.653
N721SI	13.07	1,895				Q727SM	12.81	1,857
N722SI	16.30	2,359				Q728SM	9.35	1,355
N723SI	24.34	3,529				Q729SM	14.86	2,154
N726SI	5.76	836						
Average	15.39	2,231				Average	14.30	2,073

TABLE 9. - Average tensile strength (S_t) of selected areas in graphic granite, Rockville granite, and the Calumet conglomerate: Comparison of uniaxial pull test results with Brazilian test results (All samples 4.5 mm diameter, 1.7 mm thick)

Rock	Sample	Test	Number of samples tested	Average tensile strength (S_t) psi
Graphic Granite	Quartz	Brazilian	20	2,267
do	Feldspar	do	30	2,454
do	Quartz-Feldspar Interface	do	21	1,241
Graphic Granite	Quartz	Uniaxial	12	2,009
do	Feldspar	do	29	1,074
do	Quartz-Feldspar Interface	do	19	1,012
Rockville Granite	Quartz	Brazilian	19	2,308
do	Feldspar	do	29	2,272
do	Quartz-Feldspar Interface	do	20	1,490
Rockville Granite	Quartz	Uniaxial	18	1,504
do	Feldspar	do	20	1,530
do	Quartz-Feldspar Interface	do	18	1,156
Calumet Conglomerate	Pebbles	Brazilian	32	4,358
do	Matrix	do	34	2,885
do	Pebble-Matrix Interface	do	35	2,576
Calumet Conglomerate	Pebbles	Uniaxial		
do	Matrix	do	7	2,073
do	Pebble-Matrix Interface	do	9	2,231

Although the results of the uniaxial pull tests agree qualitatively with those of the Brazilian tests, a statistical comparison of both sets of data (table 10) reveals that the strength values given by the direct pull tests are often significantly lower than the corresponding Brazilian test data. The uniaxial pull test gives interfacial adhesive strength values approximately 20 percent lower than those given by the Brazilian test.

Thus, there appears to be a bias in the Brazilian test which favors higher values of tensile strength. Although this casts doubt upon the accuracy of Brazilian tests as a method for determining absolute values of tensile strength, the results given above seem to lend credence to the use of the Brazilian test for determining approximate tensile strength and for determining the relative tensile strengths of small selected regions in rock.

A comparison of the strength of quartz-feldspar interfaces with the bulk tensile strength of a series of granites can be made with reference to table 11. This table shows the mean tensile strength of quartz-feldspar interfaces separated from graphic granite and the Rockville granite are comparable with the bulk tensile strength of a series of granites.

ANALYSIS OF DATA AND CONCLUSIONS

The data just presented shows that small selected areas containing grain boundaries can be selectively extracted from rocks and broken in the immediate vicinity of the grain boundaries to obtain a measure of the

TABLE 10. - Results of "t" test of significance for differences of means of results of direct pull and Brazilian tests of tensile strength of comparable areas in Graphic granite, Rockville granite, and Calumet conglomerate

Areas tested	t statistic	Degree of freedom	Probability that population means are significantly different	Means significantly different at 95 percent confidence level
Quartz-feldspar interfaces from graphic granite	0.91	38	0.70	No
Feldspar from graphic granite	6.32	54	>.99	Yes
Quartz from graphic granite	0.69	30	0.75	No
Quartz-feldspar interfaces from Rockville granite	1.81	36	0.96	Yes
Quartz from Rockville granite	2.63	35	0.96	Yes
Feldspar from Rockville granite	3.82	35	0.99	Yes
Pebble-matrix interfaces from Calumet conglomerate	0.46	27	0.70	No

TABLE 11. - Tensile strengths of quartz-feldspar interfaces compared with the bulk tensile strengths of six granites and the tensile strength of pebble-matrix interfaces

Rock	Interfacial strength data				Bulk granite data				
	Pull test		Brazilian test		Brazilian test		Granite	Direct pull	
	MN/m^2	psi	MN/m^2	psi	MN/m^2	psi		MN/m^2	psi
Graphic Granite	6.98	1,012	8.40	1,241	9.31	1,350	Warman		
Rockville Granite	7.98	1,156	10.50	1,478	7.80	1,130	Lac Dubbonet		
					14.00	2,000	Rainbow	9.0	1,300
					10.00	1,400	Rockville	5.6	800
					15.00	2,200	Charcoal	9.0	1,300
					7.27	1,057	Barre		

intercrystalline bonding. To the knowledge of the authors, the measurements made in this project are the first direct measurements made of the tensile strength of grain boundaries in rock. The strength of these interfaces is an important rock property because it can influence the strength and mode of failure of rock.

This research has a possible practical application in comminution research. A newly announced comminution technique, the Snyder process (13,14) reduces the grain size of ores by causing them to fracture in tension at the grain boundaries. Thus the techniques outlined above could be used to measure tensile strength of interfaces in some ores and this data might be useful in assessing the efficiency of the Snyder process in breaking the material at these interfaces.

This research has given some insight into the mechanism of intercrystalline bonding. The very fact that the crystals do not separate upon extraction from the rock indicates that the crystals are bound together at their crystalline interface. The data given above indicate that this bonding is fairly strong because quartz-feldspar interfaces can withstand stresses in excess of 1,000 psi.

The mineral pairs appear to retain the adherency in thin section (i.e. in samples that are less than 2 mm thick). Thus the mechanism responsible for intergranular adhesion evidently operates on a microscopic scale.

A detailed atomistic explanation of the mechanism responsible for this phenomenon is beyond the limits of our present knowledge, but it seems likely that the adherency is primarily a result of chemical bonding between

the mineral surfaces. Attempts have been made to minimize the effect of strengthening the interfaces through interfingering of phases by selecting straight planar interfaces. It is doubtful, however, that this effect can be completely eliminated on a microscopic scale.

The strength of any chemically bonded area which may occur far exceeds the real strength of the adjacent minerals because flaws occur in these minerals. Thus true atomic interfacial separation probably never occurs to any significant extent when mechanical forces are used to separate a pair of minerals that adhere because they have achieved atomic contact over an interfacial area.

Chemical bonds operate over very small distances. Hence, two surfaces must be brought very close together for these forces to become operative. If the mineral grains both have atomically smooth planar surfaces which were chemically bonded together, all attempts to separate them mechanically would result in the fracture in one of the minerals which had a flaw in the vicinity.

Grain boundaries in rock differ from this idealization because they are rough and contaminated and are preferred sites for cracklike cavities (15). These imperfections contribute to a greatly decreased real area of contact. Thus when a quartz-feldspar interface which has locally achieved real contact is separated mechanically, a little of the quartz remains on the feldspar and vice versa.

The determination of the fracture surface area covered by remnants may be a rough measure of the spatial extent of bonding across an interface. Figure 8 shows a bicrystal which was bonded over a large portion of the

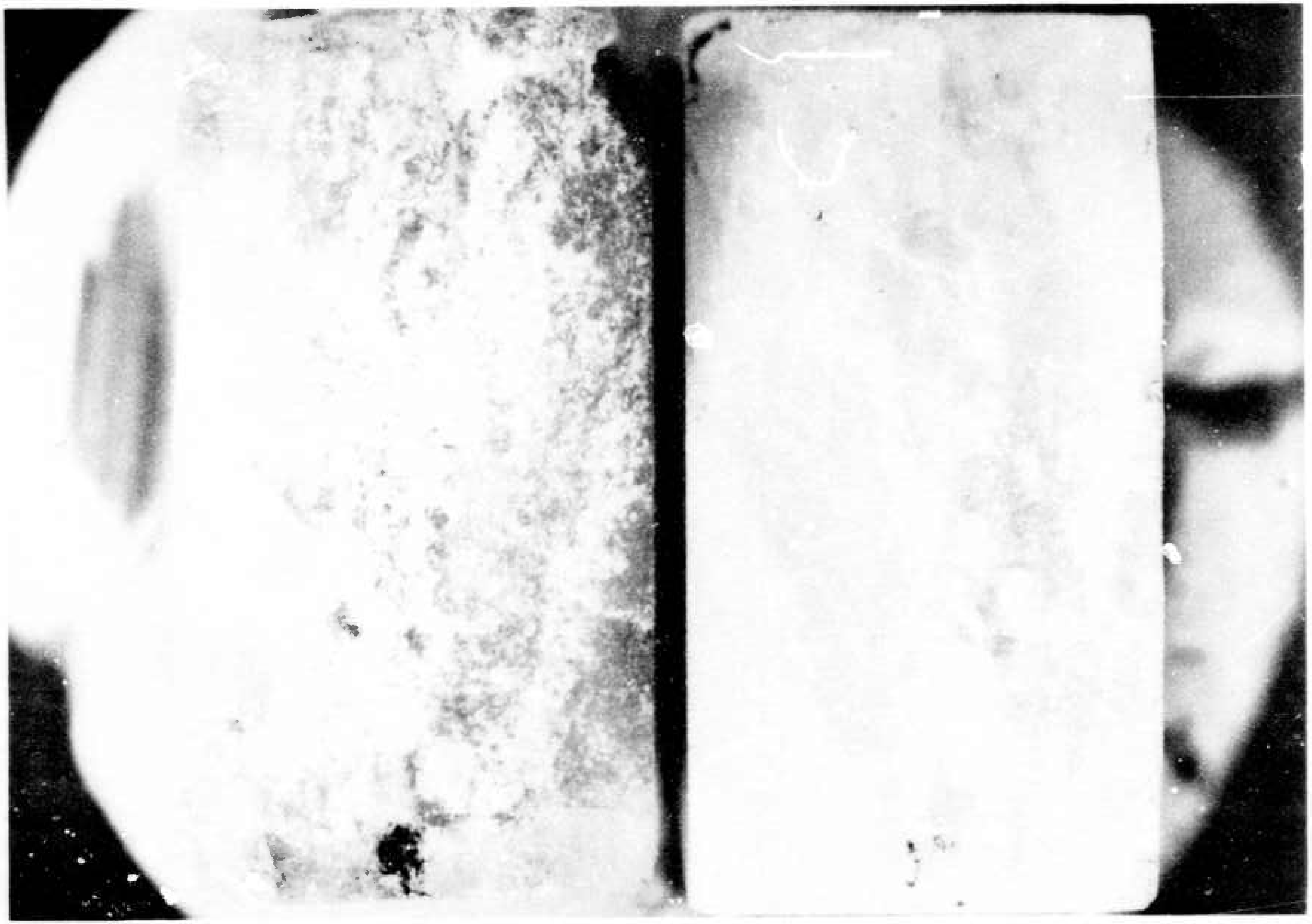

FIGURE 8. - Quartz-Feldspar Crystalline Interface With Small Proportion of Unbonded Area.

crystalline interface, whereas figure 6 showed large smooth areas which evidently were not bonded.

The most significant outcome of this research is the demonstration that small scale selected area tensile strength testing is feasible in rocks. This technique can be applied not only to intergranular adhesive strength testing but also to the determination of the strength at any selected region within the rock and hence is potentially useful in rock fragmentation research.

SUGGESTIONS FOR FURTHER RESEARCH

The phenomenon of bonding between minerals in rock has not received the attention from the rock mechanics community commensurate with its importance in the rock fragmentation process. The research reported in this report is but a small start and much more work needs to be done in this field.

The technique of selected area strength testing should be adapted to finer grained rock because most rock is finer grained than those used in this study. The direct pull test would be much better adapted for smaller samples than the Brazilian test. This coupled with the theoretical difficulties associated with the Brazilian test, dictate that future effort should be made in refining the direct pull test rather than improving the technique described above for indirect tensile testing of extracted cores.

This research program has been limited to testing the tensile strength of the grain boundaries, but a need also exists for data on the shear strength of selected areas in rock. Thus attempts should be made to develop a method of small selected area shear strength testing. A start has been

made in this project on this problem with the fabrication of a testing jig (fig. 9) consisting of a rectangular parallelepiped cut at 45 degrees to the horizontal and bisecting a cylindrical hole the size of the sample. The shear strength of the grain boundary can be determined by aligning the diameter of the sample disk containing the trace of the grain boundary parallel to the 45 degree cut and compressing the holder in a testing machine. Some preliminary tests have been conducted with this apparatus which indicate that samples can be broken in shear in this manner.

It seems reasonable to assume that the strength of a volume of the rock under a drill bit should influence the penetration of a drill into that volume. This hypothesis can be tested by performing selected area strength testing on grain boundaries and on the adjacent mineral grains in a rock. The rock could then be drilled by a small laboratory drill instrumented to output penetration rates (fig. 10). The rate of penetration into areas of interest such as grain boundaries, and single crystals could be compared with the strength data. The comparison would indicate the degree of correlation between the strength of the small areas under the drill bit and their drillability.

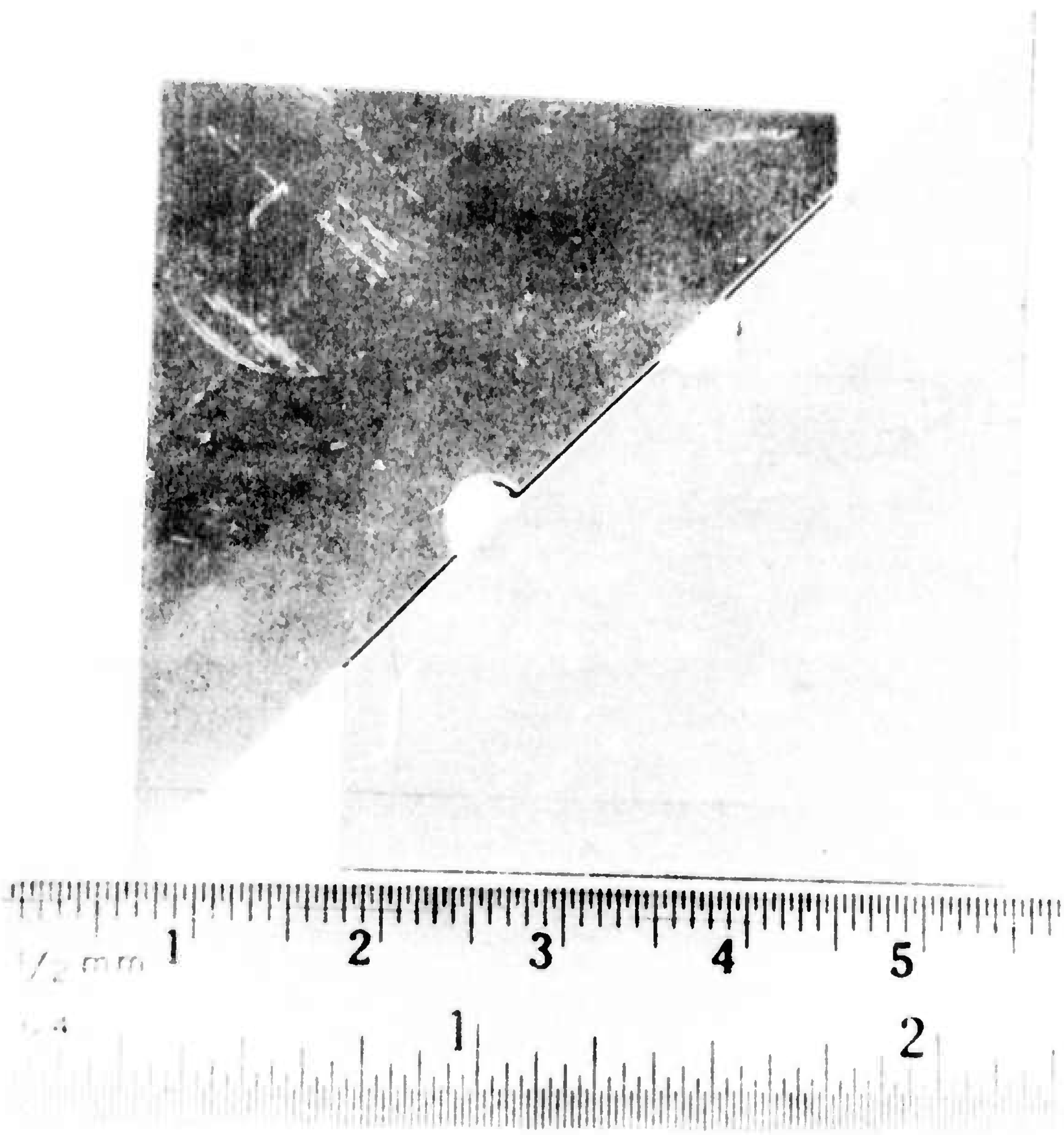

FIGURE 9. - Testing jig for shear test.

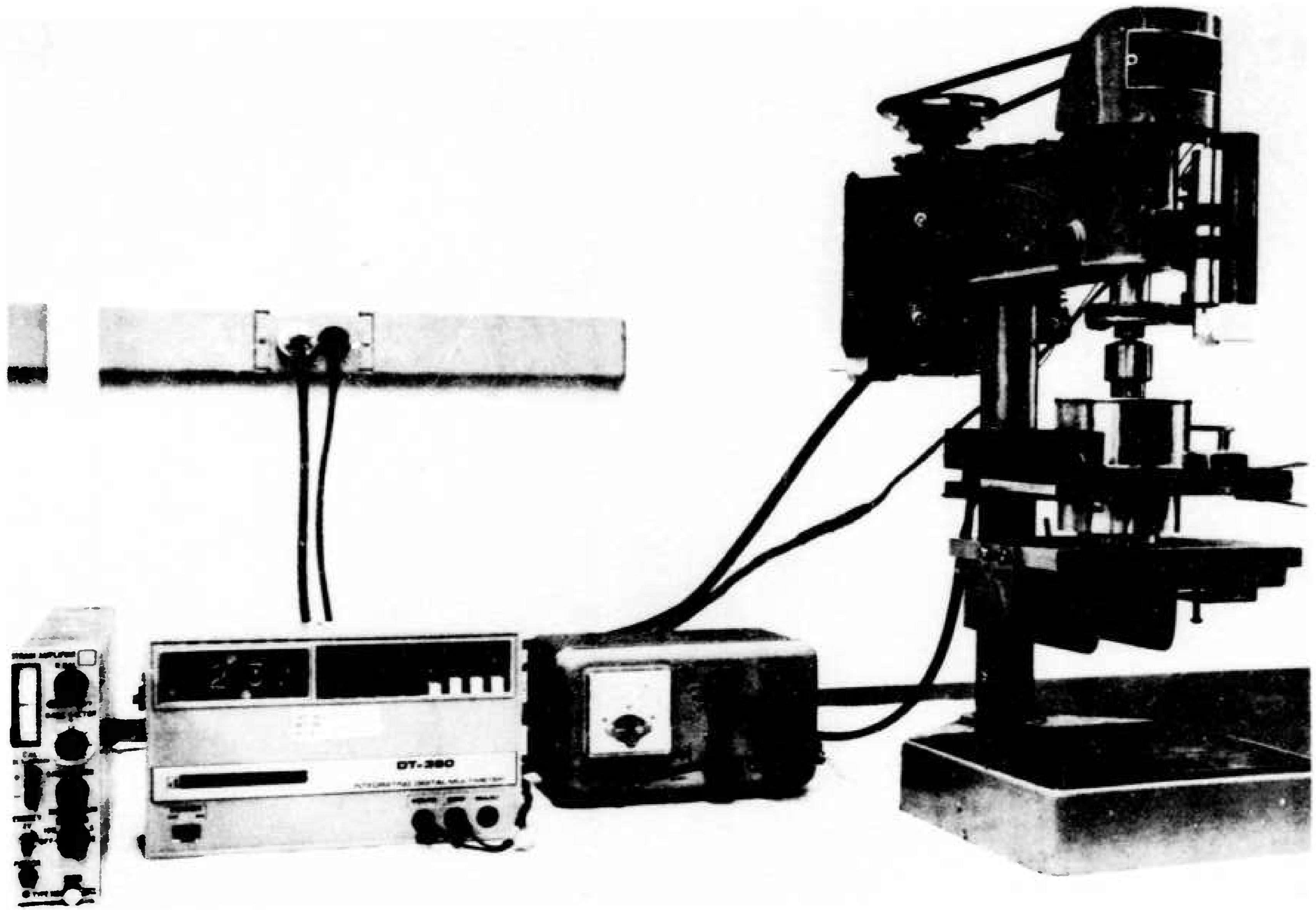

FIGURE 10. - Drill press instrumented to output penetration rates.

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